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Structure Determination of Framework-doped Tunnel Structure Manganese Oxides Using EXAFS
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Beamline(s): X18B

Introduction: Octahedral molecular sieves (OMS) have been widely used in catalysis, batteries, chemical sensors, and other applications. [1] The objective of this proposal is to study chemical states and local structure of transition metals doped in tunnel structure manganese oxides using EXAFS and EXANES. Framework doped tunnel structure manganese octahedral molecular sieves (MOMS) have exceptional catalytic properties because of the mixed valent Mn and the selectivity of doping metals. Initial work of framework doping of MOMS has been done by authors and characterization results have supported the claim. EXAFS and EXANES will be used to obtain information such as the spatial relationships of metal atoms and surrounding atoms, oxidation states of metal atoms, and the identification of possible amorphous metal oxides, which cannot be readily obtained by other techniques.

Methods and Materials: XAFS spectra at the Mn K edge and K edges of the dopants (Fe, Co and Ni) were obtained at beamline X18B. The X-rays were tuned by a Si(111) double crystal monochromators which was detuned slightly to prevent glitches from harmonics. The sample was packed into the slot of the sample holder and wrapped with Scotch tapes.

Results: EXAFS has been used to obtain local environment of Mn and doped transition metals. The atomic distances between Mn (or doped metals) and surrounding oxygen atoms have been measured. The comparison of Mn-O distance between undoped and doped OMS-2 provides information of possible structural distortion. Any change of Mn from octahedral sites to tetrahedral or other sites was directly reflected by the distance change of Mn-O bond. Since doping metal M will form its own metal oxide if the doping limit of OMS-2 is exceeded, two sets of M-O bonds have been observed because of the two different local environments (one is M in manganese oxide and the other is M in its own oxide). EXAFS obtains structural information of a short range ordered material. Amorphous metal oxides were not observed in the compounds according to the data. The above information confirms the framework doping that was limit suggested by other methods.[2]

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References:
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